

Analyte	CAS No.	Sensitivity
Reduced sulfur compounds .....	None assigned .....	Not determined.

1.2 **Applicability.** This method is applicable for the determination of emissions of reduced sulfur compounds from sulfur recovery plants where the emissions are in a reducing atmosphere, such as in Stretford units.

1.3 **Data Quality Objectives.** Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

#### 2.0 Summary of Method

2.1 An integrated gas sample is extracted from the stack, and combustion air is added to the oxygen ( $O_2$ )-deficient gas at a known rate. The reduced sulfur compounds [including carbon disulfide ( $CS_2$ ), carbonyl sulfide ( $COS$ ), and hydrogen sulfide ( $H_2S$ )] are thermally oxidized to sulfur dioxide ( $SO_2$ ), which is then collected in hydrogen peroxide as sulfate ion and analyzed according to the Method 6 barium-thorin titration procedure.

#### 3.0 Definitions. [Reserved]

#### 4.0 Interferences

4.1 Reduced sulfur compounds, other than  $CS_2$ ,  $COS$ , and  $H_2S$ , that are present in the emissions will also be oxidized to  $SO_2$ , causing a positive bias relative to emission standards that limit only the three compounds listed above. For example, thiophene has been identified in emissions from a Stretford unit and produced a positive bias of 30 percent in the Method 15A result. However, these biases may not affect the outcome of the test at units where emissions are low relative to the standard.

4.2 Calcium and aluminum have been shown to interfere in the Method 6 titration procedure. Since these metals have been identified in particulate matter emissions from Stretford units, a Teflon filter is required to minimize this interference.

4.3 Dilution of the hydrogen peroxide ( $H_2O_2$ ) absorbing solution can potentially reduce collection efficiency, causing a negative bias. When used to sample emissions containing 7 percent moisture or less, the mid-gel impingers have sufficient volume to contain the condensate collected during sampling. Dilution of the  $H_2O_2$  does not affect the collection of  $SO_2$ . At higher moisture contents, the potassium citrate-citric acid buffer system used with Method 16A should be used to collect the condensate.

#### 5.0 Safety

5.1 **Disclaimer.** This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its

use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

5.2 **Corrosive reagents.** The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water for at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burns as thermal burns.

5.2.1 **Hydrogen Peroxide ( $H_2O_2$ ).** Irritating to eyes, skin, nose, and lungs.

5.2.2 **Sodium Hydroxide ( $NaOH$ ).** Causes severe damage to eyes and skin. Inhalation causes irritation to nose, throat, and lungs. Reacts exothermically with limited amounts of water.

5.2.3 **Sulfuric Acid ( $H_2SO_4$ ).** Rapidly destructive to body tissue. Will cause third degree burns. Eye damage may result in blindness. Inhalation may be fatal from spasm of the larynx, usually within 30 minutes. May cause lung tissue damage with edema. 3 mg/m<sup>3</sup> will cause lung damage in uninitiated. 1 mg/m<sup>3</sup> for 8 hours will cause lung damage or, in higher concentrations, death. Provide ventilation to limit inhalation. Reacts violently with metals and organics.

#### 6.0 Equipment and Supplies

6.1 **Sample Collection.** The sampling train used in performing this method is shown in Figure 15A-1, and component parts are discussed below. Modifications to this sampling train are acceptable provided that the system performance check is met.

6.1.1 **Probe.** 6.4-mm (¼-in.) OD Teflon tubing sequentially wrapped with heat-resistant fiber strips, a rubberized heating tape (with a plug at one end), and heat-resistant adhesive tape. A flexible thermocouple or some other suitable temperature-measuring device shall be placed between the Teflon tubing and the fiber strips so that the temperature can be monitored. The probe should be sheathed in stainless steel to provide in-stack rigidity. A series of bored-out stainless steel fittings placed at the front of the sheath will prevent flue gas from entering between the probe and sheath. The sampling probe is depicted in Figure 15A-2.

6.1.2 **Particulate Filter.** A 50-mm Teflon filter holder and a 1- to 2-mm porosity Teflon filter (available through Saville Corporation, 5325 Highway 101, Minnetonka, Minnesota 55345). The filter holder must be